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INSTALLATION INSTRUCTIONS and TROUBLESHOOTING GUIDE

for the

OMNIPAC PAX-100 ANALYTICAL COLUMN

(10-32 End Fittings, P/N 042150)
(1/4-28 End Fittings, P/N 042165)

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INITIAL INSPECTION

As soon as you unpack the OmniPac PAX-100 Analytical Column, inspect it for shipping damage. Because many carriers limit the claim period to 10 days after the delivery date, DIONEX recommends that you report any shipping damage to the incoming carrier immediately. To request help in assessing shipping damage, or to report a missing or damaged part, contact the DIONEX Regional Office nearest you (see Section 8).

COLUMN WARRANTY

The 90-day column warranty is in effect from the day the OmniPac PAX-100 Analytical Column is shipped from DIONEX. After verifying that it was not damaged in transit, install the column in the system and test it by making several injections of the standard used to obtain the test chromatogram. The test chromatogram is shipped with the column. Contact the nearest DIONEX Regional Office (see Section 8) if the performance of the column is unsatisfactory.

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SECTION 1 - INTRODUCTION

The OmniPac PAX-100 Analytical Column (P/N 042150) was developed to greatly expand the selectivity available to the analytical chemist. The OmniPac PAX-100 allows the analytical chemist to mediate ion exchange selectivity by adding solvents to the ion exchange eluant. The addition of different solvents at different concentrations allows a broader scope of ionic analytes to be determined on a single column. The ability to use solvents on an ion exchange column also results in longer column life and less sample preparation since strongly retained hydrophobic contaminants can be eluted using solvent.

The OmniPac PAX-100 consists of a highly crosslinked, microporous core with a particle diameter of 8 μm. The ethylvinylbenzene/divinylbenzene polymeric core is produced in a manner so that the core has a reactive exterior surface to which a polymeric colloid can be attached. This polymeric colloid, or latex particles, is functionalized with a quaternary ammonium base and carries the actual anion exchange sites. The polymeric core is then coated with the polymeric colloid to create a solvent compatible ion exchange substrate.

Since the support material is polymeric-based, ionic eluants in the pH range of 0 to 14 can be used to affect selectivity and convert molecular species into ionic compounds. However, to efficiently elute ions such as alkyl and aryl amines, carboxylates and sulfonates, it is necessary to add organic solvents to the ionic eluant to prevent the organic analytes from being absorbed by the ion exchange phase and ensure the major retention mechanism is ion exchange. The highly crosslinked polymeric microporous substrate allows the use of common HPLC solvents as an eluant modifier in ion exchange separations. The OmniPac PAX-100 Analytical Column increases the flexibility of the analytical system since the user can tailor the separations to the requirements of the sample.

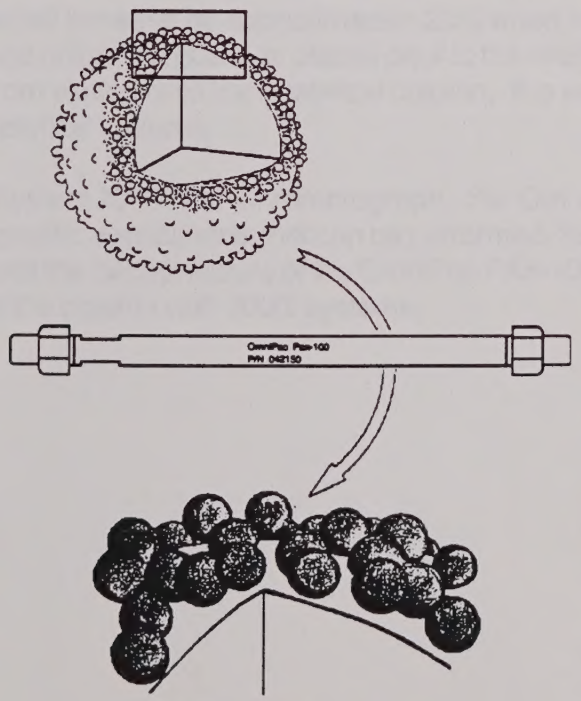

Figure 1

2.1 SYSTEM REQUIREMENTS

2.1.1 HARDWARE REQUIREMENTS

The OmniPac PAX-100 Analytical Column should be run on a DIONEX Series 4000i or 4500i Ion Chromatograph. To take full advantage of the OmniPac PAX-100, the HPLC Retrofit Assembly Kit (P/N 042940) should be ordered to upgrade your Ion Chromatograph so that your system can effectively handle eluants that contain organic solvents. The HPLC Retrofit Assembly Kit is only needed for Series 4000i and 4500i Ion Chromatographs that were purchased prior to May 1,1989. The HPLC Retrofit Kit will convert your IC system from a system that utilizes 1/4-28 thread PEEK fittings, PEEK washers, and tefzel tubing to a system that utilizes 10-32 thread ferrule-based PEEK fittings and PEEK tubing. PEEK (Polyetheretherketone) has excellent chemical resistance to most organic and inorganic liquids. Concentrated sulfuric acid, concentrated nitric acid and methylene chloride will attack PEEK. Tetrahydrofuran at concentrations of greater than 10% is not compatible with OmniPac Columns.

When performing ion exchange applications that involve a hydroxide gradient, an Anion Trap Column (ATC, P/N 037151) should be installed instead of a Gradient Mixer between the Gradient Pump and the Injection Valve. The ATC helps to minimize the baseline change due to ionic contaminants in the eluant as the ionic concentration of the eluant is increased during a hydroxide analysis.

An OmniPac PAX-100 Guard Column can be used with the OmniPac PAX-100 Analytical Column. Retention times will increase by approximately 20% when a guard column is placed in-line prior to the analytical column. A guard is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. It is easier to clean or replace a guard than it is an analytical column.

For users that have a System 2000i Ion Chromatograph, the OmniPac PAX-100 Analytical Column will have a few specific applications that can be performed. Solvent concentration and solvent viscosity can cause the backpressure of the OmniPac PAX-100 to exceed 1900 psi and therefore limit the use of the column with 2000i systems.

2.1.2 SUPPRESSOR REQUIREMENTS

Depending on your application, detection can be suppressed conductivity and/or UV/Visible spectrometry. When performing applications that require suppressed conductivity detection, an Anion MicroMembrane Suppressor (AMMS-1 P/N 037072) can be used. The AMMS-1 is compatible with all solvents at all concentrations with which the system and column are compatible.

This manual assumes that you are familiar with the installation and operation of the Ion Chromatograph and MicroMembrane Suppressor. If you do not understand the operation of your system, take the time to familiarize yourself with the operator's manuals for the products before beginning an analysis.

2.2 ELUANT STORAGE

The selectivity of the latex particles attached to the microporous substrate, has been optimized for use with hydroxide eluants. Since sodium hydroxide is converted to water in the MicroMembrane Suppressor, the concentration of hydroxide in the eluant will have only a small effect on the background conductivity. Sodium hydroxide can also be used as an eluant for applications utilizing UV detection since it easily converts molecular species into ionic compounds, thus allowing the use of an ion exchange retention mode with UV detection.

When utilizing hydroxide eluants, it is essential to keep the eluant under a helium atmosphere at all times to avoid the adsorption of carbon dioxide in the eluants. Carbon dioxide readily dissolves in dilute basic solutions producing carbonate. In the sodium hydroxide eluants that can be used with the OmniPac PAX-100 Analytical Column, the presence of carbonate in the eluant can greatly increase the baseline shift during a gradient analysis or may even affect selectivity during an analysis.

To keep eluants under a helium atmosphere at all times to minimize carbonate contamination, glass eluant reservoirs (P/N 042022) should be used since plastic reservoirs are permeable to carbon dioxide. To pressurize the eluant reservoirs with helium, an Eluant Degas Module-II (EDM-II, P/N 037124) or a modified EDM-I should be used (See Appendix A -Modifying the EDM-I). Remove the slip-on filters from the eluant line and the sparge line of each eluant reservoir. This prevents carbon dioxide adsorption on the filter when refilling the reservoirs.

Eluants containing organic solvents should be stored under a helium atmosphere and in a glass eluant reservoir. Storing eluants containing organic solvents in a pressurized glass reservoir will minimize the possibility of outgassing and carbon dioxide absorption.

2.3 INSTALLING THE ANION TRAP COLUMN

The Anion Trap Column (ATC-1, P/N 037151) is filled with high capacity anion exchange resin. The ATC-1 is used when performing hydroxide gradient applications instead of the Gradient Mixer that is usually installed between the GPM and the injection valve. When used properly, the ATC-1 will minimize the baseline shift during hydroxide gradient applications by preventing trace anionic contaminants from entering the eluant stream. To install the Anion Trap Column, complete the following steps:

A. Remove the Gradient Mixer installed between the Gradient Pump Module (GPM) pressure transducer and the injection valve.

B. Connect the GPM directly to the ATC-1. Connect a waste line to the ATC-1 outlet and direct the line to a waste container.

C. Flush the ATC-1 with 200 mL of 200 mM NaOH/5% CH_3OH at a flow rate of 2.0 mL/min.

D. Rinse the ATC-1 with the strongest eluant that will be used during the gradient analysis.

E. After flushing the ATC-1 with eluant, connect the ATC-1 to the eluant line that is connected to the injection valve.

The background conductivity of your system should be between 1.5 μS and 2.5 μS when 0.75 mM NaOH/1% CH_3OH is being pumped through the chromatographic system. The baseline shift should be no greater than 3 μS during the gradient ramp. If the baseline shifts are greater than 3 μS, the ATC-1 should be flushed using the initial operating conditions.

At the end of each operating day, the ATC-1 should be flushed to remove any impurities that may have accumulated on the ATC-1.

A. Flush the ATC-1 with 30 mL of 196 mM NaOH/1% CH_3OH (196 mM NaOH is equivalent to proportioning 98% of the 200 mM NaOH).

B. Prior to next day use of the chromatographic system, flush the ATC-1 with 30 mL of the strongest eluant of the gradient program.

2.4 USING AUTOREGEN AND ELUANTS CONTAINING SOLVENT

To minimize the baseline shift when performing an analysis that requires a hydroxide gradient, a high regenerant flow rate (10-15 mL/min) is required. To save regenerant preparation time and reduce regenerant consumption and waste, DIONEX recommends using an AutoRegen™ Accessory (P/N 039594).

However, when using an AutoRegen System to continuously remove contaminants from the regenerant and restore the regenerant to the correct state, it is necessary to replace the regenerant on a regular basis. How often the regenerant is replaced will depend on the application and the concentration of the solvent in the eluant. Minimally, the regenerant should be replaced once a week. It is not necessary to change the AutoRegen cartridge until it is completely expended.

Solvent, much like the sodium ions, passes through the membrane of the MicroMembrane Suppressor from the eluant to the regenerant stream. The solvent and the sodium ions are carried from the suppressor to the regenerant reservoir by means of the regenerant stream. As the recycled regenerant is pumped through the AutoRegen Cartridge, the sodium ions are exchanged for hydrogen ions. However, the solvent is not removed from the recycled regenerant and continues to accumulate in the recycled regenerant stream. Eventually the concentration of solvent in the recycled regenerant can cause the background conductivity to increase which can result in a noisy background. Solvent has no affect on the cartridge lifetime. The ionic strength of the eluant determines the lifetime of the AutoRegen Cartridge.

When replacing the recycled regenerant, the first 200 mL of the regenerant should be pumped to waste before recycling of the regenerant is started. Utilizing AutoRegen in this manner will allow the use of high regenerant flow rates with the minimum of consumption and waste.

3.1 GENERAL OPERATING CONDITIONS

The OmniPac PAX-100 Analytical Column has a highly crosslinked, microporous, hydrophobic core that has been agglomerated with totally permeable latex particles that are completely aminated. The latex particles carry the actual ion exchange function - a quaternary ammonium group. The polymeric structure of the packing material makes the OmniPac PAX-100 compatible with pH 0-14 eluants and organic solvents.

3.1.1 SOLVENTS

Since the microporous core has a neutral hydrophobic internal surface, it is essential that the OmniPac PAX-100 Analytical Column is operated so that eluant being pumped through the analytical column has minimally 1% organic solvent. The 1% organic solvent in the eluant will assure that the hydrophobic substrate is "wetted" and maximum column performance is maintained. The OmniPac PAX-100 can withstand all common HPLC solvents in a concentration range of 1% to 100%. However, solvents and water should be premixed in concentrations that allow proper mixing by the gradient pump and to minimize outgassing. It is therefore more practical to say that the OmniPac PAX-100 can withstand organic solvent concentrations up to 95% to ensure proper chromatographic system performance.

The ion exchange latex that has been agglomerated onto the microporous resin of the OmniPac PAX-100 Analytical Column has been optimized for use with hydroxide eluants. However, the OmniPac PAX-100 can be used with any suppressible ionic eluant that does not exceed the capacity of the MicroMembrane Suppressor. Even when performing ion exchange applications,at least 1% organic solvent must be maintained in the eluant being used. For ion exchange applications utilizing hydroxide-based eluants, acetonitrile should not be added directly to the hydroxide eluant. Best results are obtained when acetonitrile is proportioned into the eluant stream utilizing the Gradient Pump Module. When high concentrations of acetonitrile are added directly to a hydroxide eluant, hydrolysis of the acetonitrile will occur forming acetate and ammonia. Sodium hydroxide also has a limited solubility in acetonitrile. Even with this limitation, the combination of acetonitrile and sodium hydroxide are an effective eluant when proportioned together using the GPM proportioning capabilities. If proportioning CH_3CN and NaOH is not possible, eluants should be made fresh daily.

One of the benefits of the OmniPac PAX-100 Analytical Column is the ability to use most common HPLC solvents from concentrations of 1% to 95% as mobile phase modifiers in ion exchange separations. When using a solvent in an ionic eluant, column generated backpressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluant and the flow rate used. As Figure 2 shows, the column back pressure will vary as the composition of water-methanol and water-acetonitrile mixture varies.

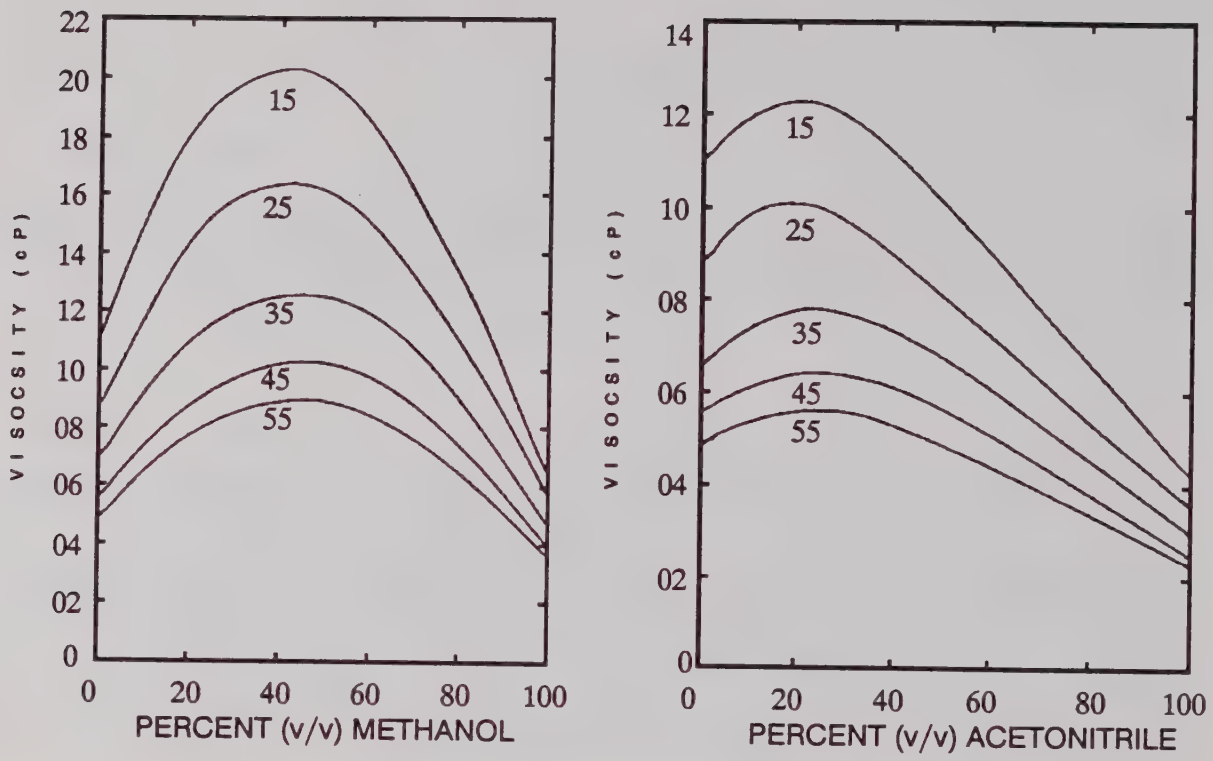

Figure 2

Variation of solvent viscosity with changing temperature and composition of water-solvent mixtures. The composition is expressed as percent (v/v) at 20.5°C and the viscosity is in centipoise. The temperature at which the viscosity was measured is indicated below each curve.*

The practical backpressure limit for the OmniPac PAX-100 Analytical Column is 5000 psi. Therefore, any combination of the above contributions to operating backpressure that totals up to 5000 psi can be used.

*C. Horvath, Ed., "High Performance Liquid Chromatography. Advances and Perspectives", Vol. 2, p. 193-194, New York, 1980.

3.1.2 GRADIENTS

Applications utilizing solvent gradients are best performed when organic solvents are premixed with deionized water. This allows proper mixing by the gradient pump to give the required gradient ramp for your chromatography. For example, if you want to run a solvent gradient from 10% solvent to 90% solvent, make the following eluants:

Eluant A: 10% solvent/90% water
Eluant B: 90% solvent/10% water

By programming the gradient pump properly, you can change from 100% Eluant A to 100% Eluant B in a prescribed time. Premixing solvents and water will prevent outgassing and refractive index problems associated with mixing neat solvents with water when using the proportioning valves of the Gradient Pump Module.

3.2 SAMPLE INJECTION

For most applications, a 10 μL injection loop will be sufficient. Generally, do not inject more than 10 nanomoles of any one analyte onto the analytical column. Injecting larger volumes of samples can result in overloading the column which can affect the detection linearity. This phenomenon will be more prevalent at higher concentrations of the analytes of interest.

Whenever practical, the chemical matrix of the sample should be matched with the eluant composition with which the column has been equilibrated at the time of injection. Matching the sample matrix will minimize baseline upsets due to refractive index and pH differences between the sample and the eluant. When matrix matching, always run a blank to ensure that no impurities are added to your sample.

3.3 CHEMICALS REQUIRED

Obtaining reliable, consistent and accurate results requires eluants that are free of ionic and spectrophotometric impurities. Chemicals, solvents and deionized water used to prepare eluants must be of the highest purity available. Low trace impurities and low particle levels in eluants also help to protect your expensive ion exchange analytical column and system components. DIONEX cannot guarantee proper column performance when the quality of the chemicals, solvents and water used to prepare eluants has been compromised.

Reagent Grade inorganic chemicals should always be used to prepare ionic eluants. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity (the universally accepted standard for reagents) should be used. These inorganic chemicals will detail the purity by having an actual lot analysis on each label.

Sodium hydroxide eluants should always be prepared from a reasonably fresh bottle of certified 50% sodium hydroxide solution that is low in carbonate (Do not use the 50% sodium hydroxide solution if a large amount of a white, sodium carbonate precipitate is present). Do not use sodium hydroxide pellets to prepare eluants since these pellets readily absorb carbon dioxide from the air.

Since solvents used with the OmniPac PAX-100 Analytical Column are added to ionic eluants to enhance the ion exchange process, the solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making "ultrahigh" purity solvents that are compatible for HPLC and spectrophotometric applications. These "ultrahigh" purity solvents will usually ensure that your chromatography is not affected by ionic impurities in your solvent. Currently at DIONEX, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima Solvents by Fisher Scientific.

The deionized water used to prepare eluants should be Type I Reagent Grade Water with a specific resistance of 17.8 megohm-cm or greater. The deionized water should be free of ionized impurities, organics, microorganisms and particulate matter larger than 0.2 μm. Bottled HPLC-Grade Water should not be used since most bottled water contains an unacceptable level of ionic impurities. Finally, thoroughly degas all deionized water prior to preparing any eluants.

3.4 COLUMN PREPARATION

CAUTION

Immediately after the installation of an OmniPac PAX-100 Analytical Column, the column must be regenerated and equilibrated as described in Section 3.4 - Column Preparation.

You MUST prepare the eluants listed below and run the accompanying gradient.

Eluant 1: Deionized water with a specific resistance of 17.8 megohm-cm or greater
Eluant 2: 90% Acetonitrile in deionized water
Eluant 3: 100 mM NaOH
Eluant 4: 40 mM NaOH/80% CH_3OH

Eluant Flow Rate: 1.0 mL/min

Time (min)	%1	%2	%3	%4
0.0	55	5	40	0
15.0	0	5	95	0
30.0	0	5	95	0
35.0	0	50	50	0
45.0	0	50	50	0
50.0	55	5	40	0
55.0	55	5	40	0
60.1	0	0	0	100
70.0	0	0	0	100

The OmniPac PAX-100 Analytical Column must then be equilibrated with eluant for 30 minutes prior to use.

3.5 SODIUM HYDROXIDE PREPARATION

The sodium hydroxide eluants that can be used with the OmniPac PAX-100 Analytical Column will readily absorb carbon dioxide, producing carbonate. Precautions must be taken during eluant preparation to minimize contamination with carbon dioxide from the air. These precautions, if taken, ensure smooth, reproducible ramps, with 1 to 3 μS total change in background conductivity.

The eluants can be prepared either volumetrically using a syringe or by weighing. Using a syringe is more effective in preventing carbonate contamination while preparing the eluants. If you decide to use the weighing method, pipette, do not pour, the 50% sodium hydroxide into the weighing dish. Minimize the time that the solution is exposed to air.

NOTE

Always degas and store all eluants in glass eluant bottles pressurized with helium. Only helium can be used to sparge and degas ionic eluants and solvents, since nitrogen is soluble in eluants.

3.5.1 SYRINGE METHOD

3.5.1.1 DEGASSING DEIONIZED WATER

A. Fill two glass eluant reservoirs (P/N 042022) with 720 mL of deionized water using a Class A volumetric flask.

1. If you are using a modified EDM-I, disconnect the vent line from each reservoir and remove the barbed fitting from the reservoir cap. Turn on the helium gas supply. Set the SYSTEM ON/OFF toggle switch of the EDM-I to ON and adjust the helium pressure to 10 psi. Set the DEGAS RATE toggle switch to HIGH. Degas the deionized water for 10 minutes before adding 50% sodium hydroxide solution.

2. If you are using a EDM-II, turn on the helium gas supply. Set the SYSTEM ON/OFF toggle switch of the EDM-II to ON. Adjust the helium pressure to 10 psi. Set the RESERVOIR ON/OFF toggle switches of the corresponding eluant reservoirs you are using for the hydroxide eluants to ON. Set the SPARGE/PRESSURE switches to the SPARGE position. Sparge each bottle for 10 minutes before adding hydroxide.

3. Vacuum degassing the deionized water is also an effective way to remove carbon dioxide. Vacuum degas the water by placing the eluant reservoir in a sonicator and drawing a vacuum on the filled reservoir with a vacuum pump. Vacuum degas the reservoir of water for 5-10 minutes. Cap each bottle and minimize the length of time the bottle is opened to the atmosphere.

3.5.1.2 ELUANT 1 PREPARATION, 0.25 mM NaOH, 16% DENATURED ETHANOL, 12% METHANOL

Using a 250 mL graduated cylinder, measure out 160 mL of denatured ethanol (Aldrich spectrophotometric grade, Cat # 27.074-1) and add to the degassed water. Using a 250 mL graduated cylinder, measure out 120 mL of methanol (Fisher brand Optima grade Cat # A454-4) and add to the ethanol/water mixture. Degass the solution for 1 minute.

Using a 25 μL Hamilton Gastight™ 1000 series syringe (Hamilton P/N 80200), inject 13 μL of the 50% sodium hydroxide solution (19.1 N) through the hole in the eluant cap when using a modified EDM-I. When using an EDM-II, remove the line from the eluant cap that is used to pressurize the eluant reservoir. Inject 13 μL of 50% sodium hydroxide through this hole.

3.5.1.3 ELUANT 2 PREPARATION, 60 mM NaOH, 28% DENATURED ETHANOL

Using a 500 mL graduated cylinder, measure out 280 mL of denatured ethanol (Aldrich spectrophotometric grade Cat # 27.074) and slowly add to the degassed water. Swirl and degass the mixture for 1 minute.

Following the procedure for preparing 0.25 mM NaOH except inject 3.2 mL of 50% sodium hydroxide (19.1 N) through the cap of the second reservoir containing 720 mL of degassed, deionized water and 280 mL ethanol. Make sure that none of the 50% sodium hydroxide solution remains on the end of the needle. Remove the syringe and quickly reinstall the appropriate fitting and lines. Minimize the time that the reservoir is opened to the atmosphere. Swirl the reservoir a few times to mix the hydroxide and the water/ethanol mixture.

3.5.2 WEIGHING METHOD

3.5.2.1 DEGASSING DEIONIZED WATER

Fill two glass eluant reservoirs with 779.48 g (E1) and 775 g (E2) of deionized water. Follow the instructions in section 3.5.1.1 to use an EDM to degas the water or vacuum degas the water by placing the eluant reservoir into a sonicator and drawing a vacuum onto the filled reservoir with a vacuum pump. Vacuum degas each reservoir of deionized water for 5 - 10 minutes. Vacuum degassing is a more convenient procedure to use when using the weighing method for eluant preparation.

3.5.2.2 ELUANT 1 PREPARATION, 0.25 mM NaOH, 16% DENATURED ETHANOL, 12% METHANOL

Measure out 125.6 g of denatured ethanol (Aldrich spectrophotometric grade, Cat # 27074-1 into a clean 250 mL graduate cyclinder. Slowly add the ethanol to the degassed deionized water. Measure out 94.9 grams of methanol (Fisher brand, Optima grade, Cat # A454) into a 100 mL graduated cyclinder. Slowly add to the ethanol/water mixture, swirl and degas for 1 minute.

Using a clean, disposable pipette, quickly measure 0.020 g of 50% sodium hydroxide into a plastic weighing dish. Quickly pour the sodium hydroxide into 1000 mL of degassed deionized water/solvent mixture. Quickly attach the cap to the reservoir and purge it with helium. Keep the time that the reservoir is open to the atmosphere as short as possible. Swirl the reservoir a few times to mix the hydroxide and water.

3.5.2.3 ELUANT 2 PREPARATION, 60 mM NaOH, 28% DENATURED ETHANOL

Measure out 219.8 g of denatured ethanol (Aldrich spectrophotometric grade, Cat # 27.074) into a 250 mL graduated cyclinder. Slowly add to the degassed deionized water. Swirl and then degas for 1 minute.

Using a clean, disposable pipette, quickly measure 4.8 g of 50% sodium hydroxide into a plastic weighing dish. Quickly pour the sodium hydroxide into 995 mL of degassed deionized water/solvent mixture. Quickly attach the cap to the reservoir and purge it with helium. Keep the time that the reservoir is open to the atmosphere as short as possible. Swirl the reservoir a few times to mix the hydroxide and water.

Obviously, these two eluants may not be the concentration of eluants required for your applications. However, following one of the above procedures will ensure that the sodium hydroxide eluants will be free of carbonate.

3.6 ELUANTS CONTAINING SOLVENTS

Applications utilizing eluants that contain solvents such as methanol and acetonitrile are straight forward as long as the solvents and water are premixed in concentrations that allow proper mixing by the gradient pump proportioning valve. Premixing water with solvent will minimize the possibility of outgassing and refractive index problems associated with using the GPM proportioning valve to mix neat solvents with water.

When mixing solvents with water remember to mix solvent with water on a volume to volume basis. If a procedure requires an eluant of 90% acetonitrile, prepare the eluant by adding 900 mL of acetonitrile to an eluant reservoir. Then add 100 mL of deionized water to the acetonitrile in the reservoir. Using this procedure to mix solvents with water will ensure that a true volume/volume eluant is obtained.

When degassing/sparging eluants containing solvents, do not degas or sparge the eluant excessively since it is possible that a volatile solvent can be "boiled" off from the solution.

3.7 REGENERANT PREPARATION

The regenerant is 20 mN sulfuric acid. Dilute 40 mL (about 40 g) of 0.50 N sulfuric acid (P/N 037164 or P/N 039601) to 1 L using deionized water. If you are not using the AutoRegen Accessory (P/N 039594), prepare several liters of the regenerant.

SECTION 4 - THEORY OF OPERATION

The OmniPac PAX-100 Analytical Column expands the selectivity control available to the analytical chemist by allowing the use of organic solvents to enhance ion exchange separations. The addition of solvents to ionic eluants allows a broader scope of ionic analytes to be determined on a single column.

The OmniPac PAX-100 key operating parameters are listed below:

Maximum Operating Pressure:	5000 psi
Organic Solvent Concentration:	1 to 100%
Eluant pH Range:	0 to 14
Maximum Eluant Flow Rate:	3.0 mL/min

The OmniPac PAX-100 should always be operated so that at least 1% organic solvent is maintained in the eluant. Maintaining 1% organic solvent in the eluant assures that the highly crosslinked hydrophobic core is "wetted" and maximum column performance is maintained. Tetrahydrofuran at concentrations greater than 10% is not compatible with OmniPac columns.

4.1 ION EXCHANGE CHROMATOGRAPHY USING THE OMNIPAC PAX-100

The OmniPac PAX-100 packing material consists of a highly crosslinked, hydrophobic microporous core with a particle diameter of 8 μm and a permeable "latex" pellicular layer which is completely aminated. The latex pellicular coating carries the actual ion exchange function - a quaternary ammonium base.

The procedure used to produce the OmniPac PAX-100 ion exchange substrate makes possible the use of solvents as a mobile phase modifier in ion exchange separations. The freedom to use solvents essentially gives the analytical chemist the ability to control the effective crosslink of the pellicular latex ion exchange phase.

Adding solvent to an ionic eluant causes the ion exchange phase to swell. The latex particles that make up the ion exchange phase have a considerably smaller diameter (0.1 μm) than the polymeric core which has a particle diameter of 8 μm. When the latex particles swell as the concentration of solvent is increased, the increase in the overall diameter of the ion exchange substrate is insignificant since the core of the substrate does not shrink or swell in the presence of solvents. However, the ion exchange phase swells as the concentration of solvent is increased in the ionic eluant. As the ion exchange phase swells, the effective crosslink of the latex is lowered since the density of aminated ion exchange polymer per unit volume decreases. The value of effective crosslink control is that it greatly expands the scope of the ion exchange material. Now separations which previously required several different columns, can be performed on a single column.

The selectivity of the ion exchange phase has been optimized for use with hydroxide eluants. When sodium hydroxide is used as an eluant, the concentration of hydroxide in the eluant has only a small effect on the background conductivity. This benefit of sodium hydroxide is especially important when performing applications that utilize an ionic gradient. Even though the ion exchange phase has been optimized for hydroxide eluants, any ionic eluant can be used to affect chromatography.

4.2 INORGANIC ANIONS

4.2.1.1 OPERATING CONDITIONS - SODIUM HYDROXIDE ELUANT

Sample Loop Volume:	10 μL
Guard Column:	OmniPac PAX-100 Guard Column
Analytical Column:	OmniPac PAX-100 Analytical Column
Eluant:	40 mM NaOH/5% CH_3OH
	or 40 mM NaOH/2.5% CH_3CN *
Eluant Flow Rate:	1.0 mL/min
Suppressor:	Anion MicroMembrane Suppressor
Regenerant:	20 mN H_2SO_4
Regenerant Flow Rate:	7-10 mL/min
Expected Background Conductivity:	2-4 μS
Expected System Operating Backpressure:	1500 - 1800 psi

* Eluant should be prepared daily or prepare 2 eluants and proportion together to minimize eluant preparation.

4.2.1.2 SAMPLE CHROMATOGRAM

1. Fluoride (2 mg/L)
2. Chloride (3 mg/L)
3. Nitrite (5 mg/L)
4. Sulfate (8 mg/L)
5. Bromide (10 mg/L)
6. Nitrate (10 mg/L)
7. Phosphate (15 mg/L)

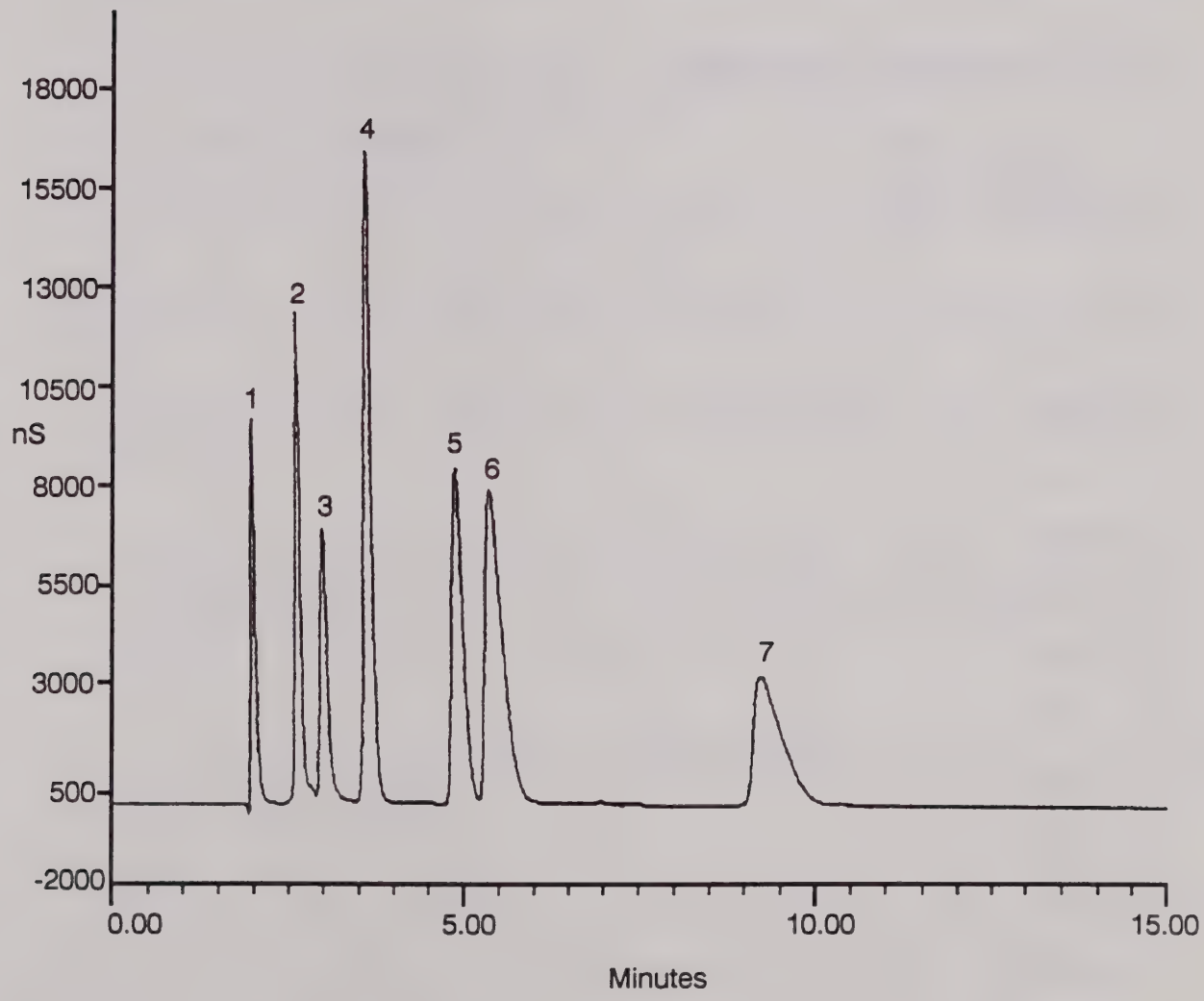

Figure 3

4.2.2.1 OPERATING CONDITIONS - CARBONATE ELUANT

Sample Loop Volume:	10 μL
Guard Column:	OmniPac PAX-100 Guard Column
Analytical Column:	OmniPac PAX-100 Analytical Column
Eluant:	3.9 mM $NaHCO_3$/3.1 mM Na_2CO_3/5% CH_3OH
Eluant Flow Rate:	1.0 mL/min
Suppressor:	Anion MicroMembrane Suppressor
Regenerant:	25 mN H_2SO_4
Regenerant Flow Rate:	5 mL/min
Expected Background Conductivity:	18 - 23 μS
Expected System Operating Backpressure:	1500 - 1800 psi

4.2.2.2 SAMPLE CHROMATOGRAM

1. Fluoride (2 mg/L)
2. Chloride (3 mg/L)
3. Nitrate (10 mg/L)
4. Phosphate (15 mg/L)
5. Sulfate (15 mg/L)

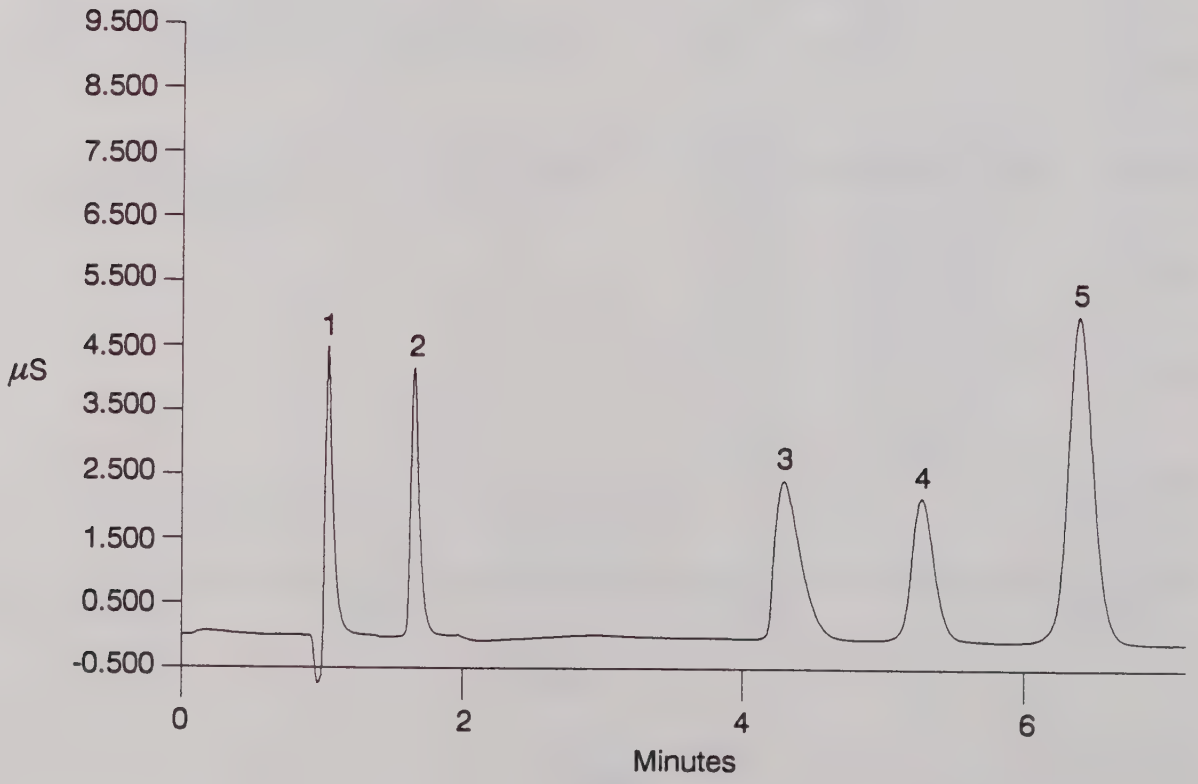

Figure 4

4.2.3.1 GRADIENT ELUTION OF INORGANIC AND ORGANIC ANIONS

The sodium hydroxide concentration of the eluant at the beginning of the gradient program, is weak enough that fluoride elutes after the void volume. The weak ionic eluant will also separate several weakly retained monovalent organic acids. The sodium hydroxide concentration is increased to elute polyvalent ions such as trivalent phosphate and citrate. See Section 3 - Operation - for eluant preparation instructions.

Equilibrate the column thoroughly with 0.25 mM NaOH/12% methanol/16% ethanol before injecting the sample. Equilibration time is typically 15 minutes. If the final eluant concentration used in a gradient is stronger than the one shown in the example chromatogram (i.e. 60% mM NaOH/28% ethanol) , the time required to equilibrate the OmniPac PAX-100 should be increased.

If an injection is made before the column is fully equilibrated with the weak eluant, the early eluting peaks (fluoride and the monoprotic organic acids) will elute too soon and resolution will be impaired. Furthermore, retention times will not be reproducible.

If better resolution is needed for the first eluting peaks, dilute eluant E1, 1 mM NaOH, since this part of the chromatogram is run isocratically at an eluant concentration of 0.25 mM NaOH.

The Gradient shown in the example can be adjusted to improve resolution or adjust retention times either by changing the gradient timing or by changing the eluant gradient proportions.

A. Keep the concentrations of Eluant 1 and Eluant 2 constant and adjust the gradient time. This is the simplest way to compensate for total system differences if resolution is the problem.

   For example, if nitrate and sulfate are well resolved but phosphate and bromide are not, multiply the gradient program times by a factor less than 1 (e.g., 0.90) to increase the gradient slope. On the other hand, if nitrate and sulfate are coeluting, multiply the gradient program times by a factor greater than 1 (e.g, 1.1) to have a gradient slope that is less steep.

   To reduce the total gradient time, and if resolution allows it (i.e., not all the peaks shown in the sample chromatogram are present in the sample), multiply the gradient program time by a factor less than 1.

B. Change the proportions of Eluant 1 and Eluant 2 and adjust the gradient program times. This approach requires more time to develop and more knowledge in methods development work. The advantage of this approach is that a method can be tailored for a particular application, where selectivity, resolution, and the total run time are optimized. Be aware that changing the gradient can affect the elution order of ions of different charge. For example, increasing the gradient ramp slope will cause sulfate to elute earlier than nitrate.

If resolution is a problem, consider these possibilities before changing the gradient to improve resolution:

1. Make sure that eluants E1 and E2 have been prepared correctly. Too low a hydroxide concentration in one or both eluant results in poor resolution of phosphate and bromide. Too high a concentration in one or both eluants results in poor resolution of nitrate and sulfate.

2. Check the eluant flow rate. If the flow rate is greater than 1.0 mL/min, resolution of phosphate and bromide may suffer. If the flow rate is less than 1.0 mL/min, resolution of nitrate and sulfate may improve.

3. The column capacity may differ slightly from that of the column used to obtain the sample chromatogram. In this case it may be necessary to adjust the gradient to provide the desired resolution.

4. The concentration of CH_3OH can be varied during the analysis to affect resolution. By increasing the concentration of CH_3OH, it is possible to obtain better resolution of weakly retained anions. However, some loss in resolution of the later eluting anions may be seen.

4.2.3.2 OPERATING CONDITIONS

Sample Loop Volume:		10 μL
Guard Column:		OmniPac PAX-100 Guard Column
Analytical Column:		OmniPac PAX-100 Analytical Column
Eluants,	Eluant 1:	0.25 mM NaOH/12% methanol/16% ethanol
	Eluant 2:	60 mM NaOH/28% ethanol
Eluant Flow Rate:		1.0 mL/min
Suppressor:		Anion MicroMembrane Suppressor
Regenerant:		20 mN H_2SO_4
Regenerant Flow Rate:		15 mL/min
Expected Background Conductivity:		0.25 mM NaOH; 2-4 μS
		60 mM NaOH; 4-7 μS
Expected System Operating Backpressure:		2000 - 2300 psi

Gradient Program

Time (min)	%1	%2	%3	%4	V5	V6	Comments
0.0	100	0	0	0	0	0	Equilibration
10.0	100	0	0	0	1	0	load
10.1	100	0	0	0	1	0	inject
13.1	100	0	0	0	1	0	isocratic condition
30.0	70	30	0	0	1	0	gradient
45.0	0	100	0	0	1	0	end gradient
50.0	0	100	0	0	1	0	
50.1	100	0	0	0	1	0	gradient reset

NOTE

10 minutes are required for equilibration of the column with the weak eluant prior to injecting the next sample.

4.2.3.3 SAMPLE CHROMATOGRAM

1. Fluoride
2. Acetate
3. Propionate
4. Lactate
5. Quinate
6. Formate
7. Pyruvate
8. Monochloroacetate
9. Chloride
10. Nitrite
11. Dichloroacetate
12. Mannuronate
13. Nitrate
14. Selenite
15. Succinate
16. Sulfite
17. Malate
18. Tartrate
19. Sulfate
20. Phthalate
21. Phosphate
22. Chromate
23. Citrate

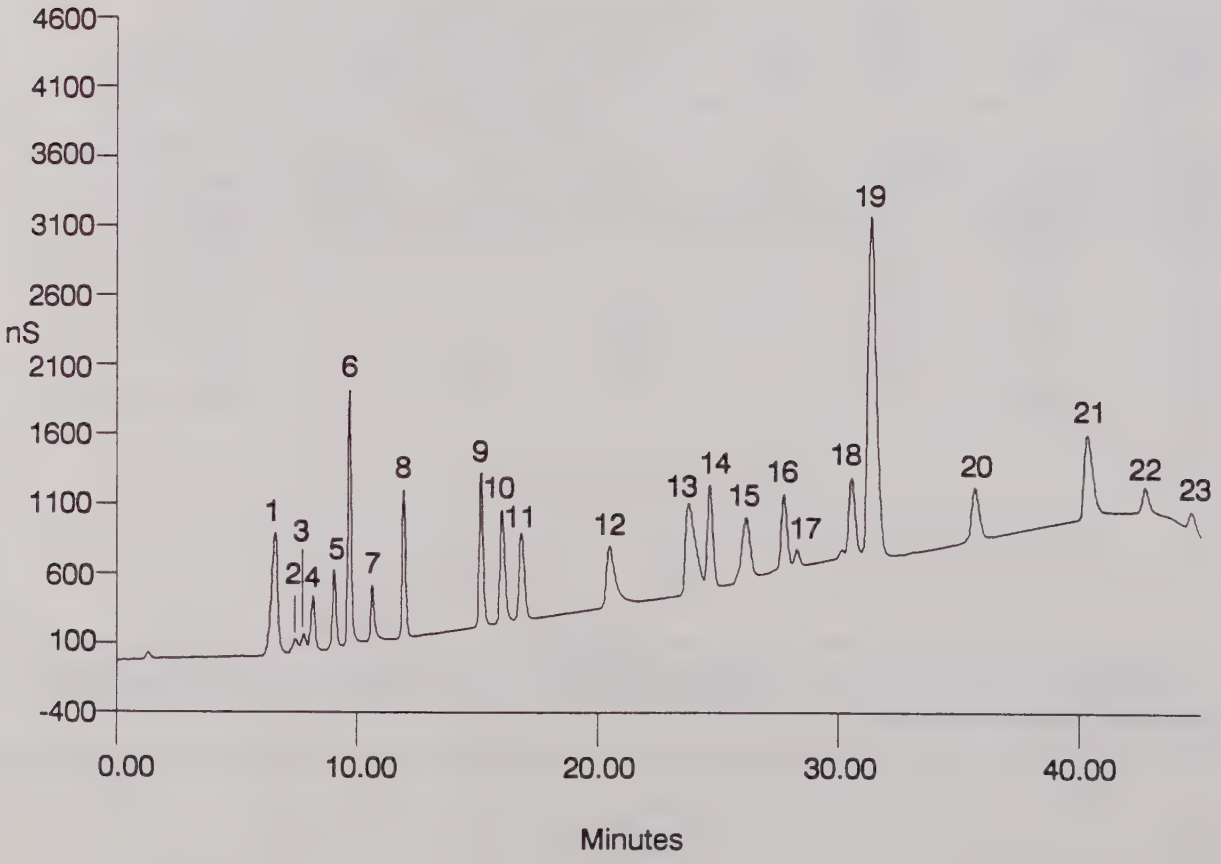

Figure 5

4.3 SEPARATION OF AROMATIC ACIDS

Using UV detection and the OmniPac PAX-100 Analytical Column, it is possible to separate, by ion exchange, a wide variety of aromatic acids which cannot be separated by ion suppression or ion pair. The uniform peak width and uniform peak asymmetry gives an indication of the excellent focusing properties using the ionic gradient.

4.3.1 OPERATING CONDITIONS

Sample Loop Volume:		10 μL
Guard Column:		OmniPac PAX-100 Guard Column
Analytical Column:		OmniPac PAX-100 Analytical Column
Eluants,	Eluant 1:	Deionized Water having a specific resistance of 17.8 megohm-cm or greater
	Eluant 2:	80% CH_3CN
	Eluant 3:	1 M NaCl/4 mM NaOH
Eluant Flow Rate:		1.0 mL/min
Detection:		UV at 254 nm
Expected System Operating Backpressure:		1800 - 2000 psi

Gradient Program

Time (min)	%1	%2	%3	Comments
0.0	70	25	5	Equilibration with weak eluant
10.1	70	25	5	Injection of Sample
30.0	35	25	40	Gradient Ramp

4.3.2 SAMPLE CHROMATOGRAM

1. Benzoate
2. Benzenesulfonate
3. Toluenesulfonate
4. p-Chlorobenzenesulfonate
5. p-Bromobenzoate
6. 3,4-Dinitrobenzoate
7. Phthalate
8. Terephthalate
9. p-Hydroxybenzenoate
10. p-Hydroxybenzenesulfonate
11. Gentisate
12. Trimesate
13. Pyromellitate

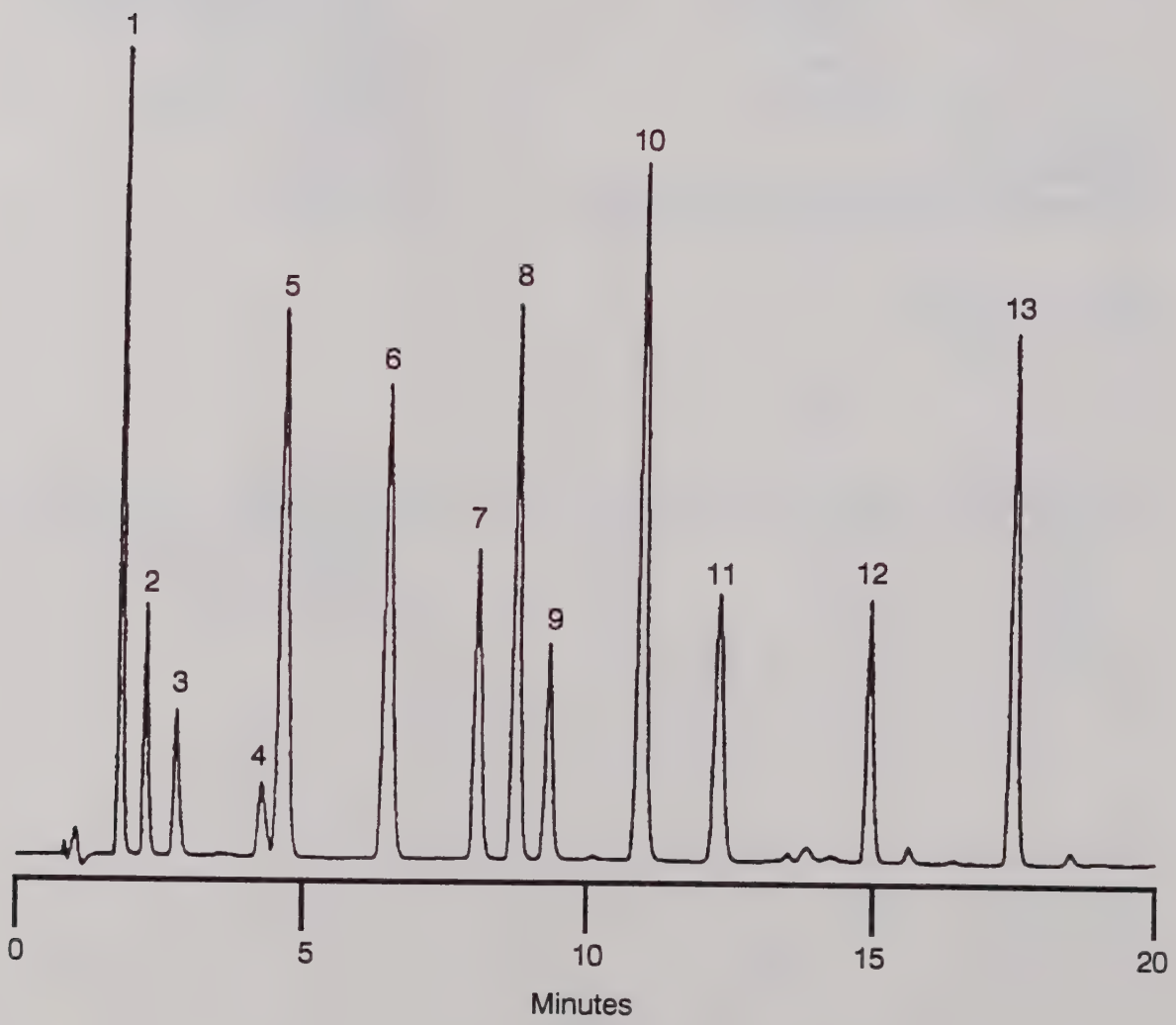

Figure 5

SECTION 5 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the OmniPac PAX-100 Analytical Column. For more information on problems that originate with the Ion Chromatograph (IC) or the MicroMembrane Suppressor, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, call the DIONEX Regional Office nearest you (see Section 8).

5.1 HIGH BACKPRESSURE

Total system backpressure when using the OmniPac PAX-100 Analytical Column at 1.0 mL/min should be less than 3000 psi. Refer to Section 3.1 - Solvent Viscosity - to see how solvent concentration can affect the column operating backpressure. If the backpressure is higher than 3000 psi, it is advisable to find out what is causing the high pressure. The system should be used with an in-line filter for the eluants. Make sure you have one in place and that it is not contaminated.

A. Make sure that the pump is set to 1.0 mL/min. Higher flow rates will cause higher pressure. Measure the pump flow rate if necessary.

B. Find out what part of the system is causing the high backpressure. It could be a piece of tubing that has plugged or whose walls are collapsed, an injection valve with a plugged port, a column with particulates plugging the bed support, a plugged high-pressure in-line filter, the MicroMembrane Suppressor or the detector.

   To find out which part of the chromatographic system is causing the problem, disconnect the pump eluant line from the injection valve and turn the pump on. Watch the pressure; it should not exceed 50 psi. Continue adding the system's components (injection valve, column, MicroMembrane Suppressor, detector) one by one, while watching the pressure. The pressure should increase up to a maximum of 3000 psi when the column is connected. The MicroMembrane Suppressor will add up to 100 psi. No other components should add more than 100 psi of pressure. Refer to the appropriate manual for cleanup or replacement of the problem component.

C. If the analytical column is the cause of high backpressure, its inlet bed support may be contaminated. To change the bed support, follow the instructions below using one of the two spare bed supports included in the Ship Kit.

   1. Disconnect the column from the system.

   2. Using two open-end wrenches, carefully unscrew the inlet (top) column end fitting.

   3. Turn the end fitting over and tap it against a bench top or other hard, flat surface to remove the bed support and seal assembly. Discard the old assembly.

4. Place a new bed support assembly into the end fitting. Use the end of the column to carefully push the bed support assembly into the end fitting.

	OmniPac PAX-100 Columns with 10-32 End Fittings (P/N)	OmniPac PAX-100 Columns with 1/4-28 End Fittings (P/N)
Analytical Column	042150	042165
Guard Column	042151	042166
Bed Support Assembly	042955	042310
Seal Washer	042956	039835
Bed Support	041375	041375
End Fitting	042367	041353

5. Screw the end fitting back onto the column. Tighten it fingertight, then an additional 1/4 turn (25 in x lb). Tighten further only if leaks are observed.

NOTE

If any of the column packing becomes lodged between the end of the column and the bed support washer assembly, no amount of tightening will seal the column. Make sure that the washer and the end of the column are clean before screwing the end fitting back onto the column.

6. Reconnect the column to the system and resume operation.

5.2 HIGH BACKGROUND, NOISE

In a properly working system, the background conductivity level for the standard eluant system is shown below:

ELUANT	EXPECTED BACKGROUND CONDUCTIVITY
0.75 mM NaOH/2% CH_3OH	1-3 μS
80 mM NaOH/2% CH_3OH	4-7 μS
40 mM NaOH/5% CH_3OH	2-4 μS

The background conductivity typically increases between 1 and 3 μS when running a gradient as described in Section 4.

A. Make sure that the eluants and the regenerant are made correctly. Were the proper precautions taken to prepare the sodium hydroxide eluant? If carbonate was present in the eluant, the Anion Trap Column will eventually be spent and the background level will increase.

B. Make sure that the eluants are made from chemicals with the recommended purity.

C. Make sure that the deionized water used to prepare the reagents has a specific resistance close to 17.8 megohm-cm or greater.

D. Has the Anion Trap Column (ATC-1) been installed correctly? If it has not, install one as directed in Section 2.3 and watch the background conductivity. If the background conductivity is now low, this means that the ATC-1 is trapping contaminants from the eluant. The eluants probably have too many impurities (see items 1 - 3 above).

   If the ATC-1 is already installed, remove it. Is the background conductivity still high? If the background conductivity decreases, the ATC-1 is the source of the high background conductivity.

   1. Disconnect the ATC-1 from the analytical column and direct the outlet to waste.

   2. Pump approximately 250 mL of 196 mM NaOH/1% CH_3OH through the ATC-1 at 2 mL/min.

   3. Equilibrate the ATC-1 at 1 mL/min for 30 minutes with the strongest eluant used during the gradient run.

   4. If the problem persists, replace the ATC-1.

E. Remove the OmniPac PAX-100 Analytical Column from the system. Is the background conductivity still high? If the column is the cause of the high background conductivity, clean the column as instructed in Section 6.4.

F. To eliminate the hardware as the source of the high background conductivity, bypass the MicroMembrane Suppressor and pump deionized water with a specific resistance of 17.8 megohm-cm or greater through the system. The background conductivity should be less than 2μS. If it is not, check the detector/conductivity cell by injecting deionized water directly into it.

G. If the above items have been checked and the problem persists, the suppressor is probably causing the problem.

1. Check the regenerant flow rate at the REGEN OUT port of the MicroMembrane Suppressor. This flow should be greater or equal to 12 mL/min.

2. Check the eluant flow rate. It should be 1.0 mL/min.

3. Prepare fresh regenerant solution. Bypass the Anion AutoRegen Regenerant Cartridge (if you are using the AutoRegen Accessory). If the background conductivity is high, you probably need to clean or replace your MicroMembrane Suppressor. Refer to the MicroMembrane Suppressor Installation Instructions and Troubleshooting Guide for assistance.

4. If you are using an AutoRegen Accessory, connect the freshly prepared regenerant to the Anion AutoRegen Regenerant Cartridge. Pump approximately 200 mL of regenerant through the Anion AutoRegen Regenerant Cartridge to waste before recycling the regenerant back to the regenerant reservoir. If the background conductivity is now high, you probably need to replace the Anion AutoRegen Regenerant Cartridge (P/N 039564). Refer to the AutoRegen Regenerant Cartridge Refill Installation Instructions and Troubleshooting Guide for assistance.

5.3 POOR PEAK RESOLUTION

Poor peak resolution can be due to:

A. Loss of Column Efficiency:

1. Check to see if headspace has developed in the analytical column (e.g., due to improper use of the column such as using the column without 1 % organic solvent in the eluant or submitting it to high pressures). Remove the column's top end fitting (see Section 5.1, Step C). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed, creating a headspace. The column must be replaced.

2. Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient. Make sure you are using tubing with an ID of no greater than 0.012 inch, in all cases, between the injection valve and the detector cell inlet, and that the tubing lengths are as short as possible. Check for leaks.

B. Shorter Retention Times - Peaks elute too fast, compromising resolution:

1. Check to see if eluant flow rate is faster than 1.0 mL/min. Check the eluant flow rate after the column.

2. Check to see if the eluants' composition and concentration are correct. An eluant that is too strong will cause the peaks to elute sooner. Prepare fresh eluant. If you are using a GPM to proportion the eluant components from two or three different eluant reservoirs, the resulting eluant composition may not be accurate enough for the application. Use one reservoir containing the correct eluant composition to see if this is the problem. This may be a problem when one of the proportionized eluants is less than 5%.

3. Column contamination can lead to a loss of column capacity because all of the anion exchange sites will no longer be available for the sample ions. Polyvalent anions might be concentrating on the column. Refer to Section 6.4 for recommended column cleanup procedures.

   Possible sources of column contamination are impurities in chemicals and in the deionized water used. Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of at least 17.8 megohm-cm.

C. Diluting the eluant will improve peak resolution, but will also increase the analytes' retention times. If a 10% dilution of the eluant is not sufficient to obtain the desired peak resolution, or if the resulting increase in retention times is unacceptable, clean the column (see Section 6.4).

   After cleaning the column, reinstall it in the system and let it equilibrate with eluant for about 30 minutes. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment, since the contaminants should be eluted from the column.

D. If poor resolutions and efficiencies are observed for the very early eluting peaks near the system void volume compared to the later eluting peaks, check the following:

1. Improper eluant E1 concentration may be the problem. Remake the eluant as described in Section 3.

2. Column overloading may be the problem. Reduce the amount of sample ions being injected onto the analytical column by either diluting the sample or injecting a smaller volume onto the column.

3. Improperly swept out volumes anywhere in the system prior to the analytical column may be the problem. See item A above.

4. Sluggish operation of the injection valve may be the problem. Check the air pressure and make sure there are no gas leaks or partially plugged port faces. Refer to the valve manual for instructions.

5.4 SPURIOUS PEAKS

A. Run the gradient program without making an injection. Examine the baseline. If you see spurious peaks, the column may be contaminated.

If the samples contain an appreciable level of polyvalent ions and the column is used with a weak eluant system, polyvalent anions may be contaminating the analytical column. The retention times for the analytes will then decrease and spurious, inefficient (broad) peaks can show up at unexpected times. Clean the column as indicated in Section 6.4. Using the recommended eluant will ensure that strongly retained polyvalent anions are eluted before the next injection.

B. Run the gradient program again, this time switching the injection valve but not injecting sample or standard (make sure that the sample loop contains either deionized water or eluant). If you see a baseline upset, especially at the beginning of the chromatogram, it is probably due to the injection valve.

When an injection valve is actuated, the possibility of creating a baseline disturbance exists. This baseline upset can show up as a peak of varying size and shape. It will happen when the injection valve needs to be cleaned or retorqued (see valve manual). Check to see that there are no restrictions in the tubing connected to the valve. Also check the valve port faces for blockage and replace them if necessary. Refer to the Valve Manual for troubleshooting and service procedures. Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantitation of the peaks of interest.

If cleaning and retorquing the valve does not help, replace the valve. Use a DIONEX High Pressure Injection Valve (P/N 037142) or a DIONEX High Pressure Inert Valve (P/N 037143) as required.

6.1 RECOMMENDED OPERATION PRESSURES

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for the OmniPac PAX-100 Analytical Column is 5000 psi.

6.2 COLUMN START-UP

The column is shipped in 100 mM NaOH/5% CH_3OH storage solution.

Prepare the eluant shown on the test chromatogram, install the column in the chromatography module and test the column performance under the conditions described in the test chromatogram. Let the column equilibrate with eluant for a few minutes. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

6.3 COLUMN STORAGE

The column's storage solution should be eluant. If the column will not be used for one week or more, prepare it for long-term storage. Pump the column down using the base-soluble/organic contaminants gradient program described in Section 6.4.2. Flush the column for 10 minutes with 100 mM NaOH/5% CH_3OH. Cap both ends securely, using the plugs supplied with the column.

6.4 COLUMN CLEANUP

6.4.1 POLYVALENT ANIONS AND ACID-SOLUBLE CONTAMINANTS

A. Prepare a 500 mL solution of 1 M HCl/0.1 M KCl/5% CH_3OH.

B. Disconnect the analytical column from the injection valve and the MicroMembrane Suppressor. Disconnect the Gradient Mixer or the ATC-1 from the Gradient Pump Module (GPM). Connect the OmniPac PAX-100 Analytical Column directly to the GPM. Direct the effluent from the analytical column directly to waste.

C. Set the pump flow rate to 1.0 mL/min.

D. Pump 1 M HCl/0.1 M KCl/5% CH_3OH solution through the column for 30-60 minutes.

NOTE

Nitric acid should not be used instead of hydrochloric acid since nitric acid will not efficiently remove iron contaminants.

E. Pump 100 mM NaOH/5% CH_3OH through the column for 15 minutes.

F. Reconnect the suppressor to the analytical column.

G. Equilibrate the column with eluant before resuming normal operation.

6.4.2 BASE-SOLUBLE AND ORGANIC CONTAMINANTS

A. Disconnect the suppressor from the analytical column. Direct the effluent from outlet line of the analytical column to waste.

B. Set the pump flow rate to 1 mL/min. Use the following gradient program to remove anionic and organic contaminants from the OmniPac PAX-100 Analytical Column.

Eluant 1: Deionized Water
Eluant 2: 90% Acetonitrile
Eluant 3: 100 mM NaOH
Eluant Flow Rate: 1.0 mL/min

Time (min)	%1	%2	%3
0.0	55	5	40
15.0	0	5	95
25.0	0	5	95
30.0	0	50	50
40.0	0	50	50
45.0	55	5	40 Hold for 5 minutes

C. Reconnect the suppressor to the analytical column.

D. Equilibrate the column with eluant before resuming normal operation.

NOTE

It is not necessary to use sodium hydroxide and acetonitrile in the above cleanup procedure. You may find it more beneficial, depending on you sample matrix, to use a different ionic eluant such as sodium chloride and a different solvent such as isopropyl alcohol.

6.4.3 CARBONATE REMOVAL

Carbonate should not really be considered a column contaminant, but the presence of carbonate on the OmniPac PAX-100 Analytical Column or in the eluant when utilizing hydroxide eluants to perform chromatography can cause shifts in retention times, irreproducible results, high background conductivity and large baseline shifts. Following the recommended procedures for eluant preparation and storage will minimize the chances of hydroxide eluants absorbing carbon dioxide, thereby producing carbonate and affecting chromatography. The following steps can be used to remove carbonate from the PAX-100 Analytical Column.

A. Connect the OmniPac PAX-100 Analytical Column directly to the Gradient Pump Module (GPM), bypassing the Gradient Mixer or the Anion Trap Column (ATC-1). Direct the column effluent directly to waste bypassing the MicroMembrane Suppressor.

B. Prepare an eluant of 200 mM NaOH/5% CH_3OH, taking the necessary precautions to minimize contamination with carbon dioxide. See Section 3 for complete details.

C. Pump 250 mL of the 200 mM NaOH/5% CH_3OH through the column at 1.0 mL/min.

D. Rinse the analytical column with eluant for 30 minutes. If performing a gradient application, rinse the column with the strongest eluant used during the gradient ramp.

E. When performing gradient applications, equilibrate the analytical column with the weakest eluant. Continue by pumping eluant through the column until the background conductivity stabilizes. The column is fully equilibrated when successive injections of a standard give reproducible retention times.

SECTION 7 - INSTALLATION OF DIONEX LIQUID LINE FITTINGS

During the course of installing and using the OmniPac PAX-100 Analytical Column with the Anion MicroMembrane Suppressor (AMMS-1), it will be necessary to assemble a new Tefzel® connecting liquid line with a 10-32 PEEK bolt and ferrule assembly on one end, for connection to the OmniPac PAX-100 Analytical Column, and a ThermoFlare 1/4-28 fitting on the other end, for connection to the AMMS-1. The following assembly procedures outline the assembly of these liquid line fittings on Tefzel tubing.

7.1 INSTALLING THERMOFLARE FITTINGS

DIONEX ThermoFlare fittings are used for all tubing connections made with 0.012" ID Tefzel tubing throughout the liquid flow path on the Series 2000i and 4000i Ion Chromatographs except on the high pressure end of the chromatograph when eluants containing solvents are being pumped at pressures above 2000 psi. Tefzel tubing with ThermoFlare fittings are used after the analytical column and throughout the low pressure end of the chromatograph in all applications. Tefzel tubing with ThermoFlare fittings offer important advantages for use in low pressure ion chromatography.

A. Tefzel tubing with ThermoFlare fittings withstand pressures up to 4500 psi using aqueous eluants or 2000 psi using reverse phase solvents for eluants.

B. They contain no metal.

C. The tubing cannot back out of the fitting and create unwanted mixing void volumes.

D. The flare can be remade up to 100 times at no additional cost with minimal tubing loss when aqueous solvents are used.

CAUTION

Do not remake the flare after the tubing has been used with acetonitrile, tetrahydrofuran or methanol.

If you already have a connecting liquid line for use between an analytical column with 1/4-28 end fittings and a Anion MicroMembrane Suppressor with ThermoFlare end fittings on both ends, you may choose to cut off one of the ThermoFlare end fittings with a razor blade and replace it with a 10-32 PEEK bolt and ferrule for connection to the OmniPac PAX-100 Analytical Column. After cutting off one of the ThermoFlare end fittings, proceed to Section 7.2 for instructions on assembling the 10-32 PEEK bolt and ferrule on the Tefzel line.

The sealing surface of the fitting is made by first heating and then flaring the end of the tubing. A special flaring tool (120 VAC version, P/N 037196 or 230 VAC version, P/N 037197) is required to make ThermoFlares. Complete instructions for using the tool are in Document No. 032843.

If you prefer not to buy a flaring tool, tubing with factory-made ThermoFlares is available from DIONEX in the following IDs and lengths:

0.012" ID Tubing

4" long (P/N 039335), each
8" long (P/N 039336), each
12" long (P/N 039337), each
24" long (P/N 039338), each

0.020" ID Tubing

4" long (P/N 039339), each
16" long (P/N 039340), each
72" long (P/N 039341), each

0.037" ID Tubing

12" long (P/N 039342), each
36" long (P/N 039343), each

7.2 INSTALLING 10-32 PEEK FITTINGS

The DIONEX Ferrule Fitting System consists of a length of 1/16" OD tubing, a 10-32 threaded tapered port, a HPLC 10-32 threaded fitting bolt (P/N 043275) and a double-cone ferrule (P/N 043276) as shown in Figure 7.

Although the DIONEX Ferrule Fitting System can be used on any semi-rigid or rigid tubing, including Tefzel, PEEK or stainless steel tubing with an outside diameter of 0.062", DIONEX recommends the use of Tefzel tubing to make the eluant line between analytical columns with 10-32 PEEK end fittings and the Anion MicroMembrane Suppressor (AMMS-1) because of its chemical resistance.

The internal diameter of the tubing must be small enough to prevent the wall of the tubing from collapsing when the fitting is installed. An internal diameter of 0.012" is recommended for all liquid lines in the eluant flow path.

The fitting is intended to be installed into a 10-32 threaded, tapered port as shown in Figure 7 below. The fitting is fabricated from high-strength PEEK plastic which conforms to a relatively large range of port geometries. The fitting, however, works most reliably in the DIONEX 10-32 PEEK ports.

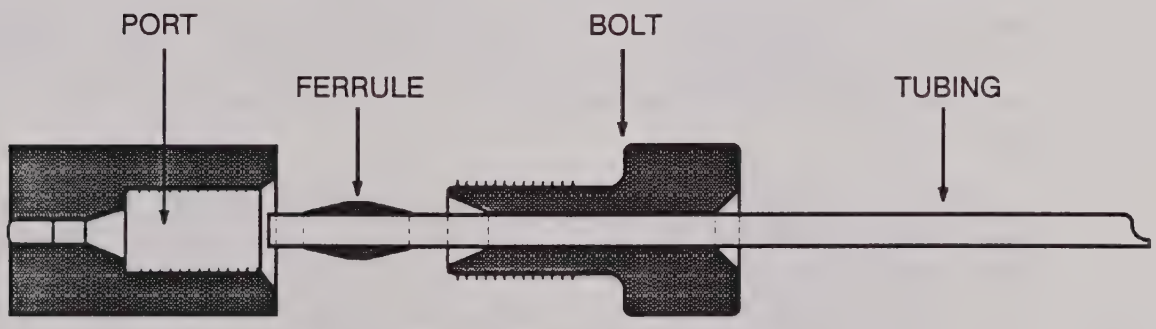

Figure 7
10-32 PEEK Ferrule Fitting System

The following procedure is used to install the 10-32 PEEK bolt and ferrule fittings on liquid lines. For the most reliable performance, DIONEX recommends the use of Tefzel tubing (because of its chemical resistance) as the connecting tubing between analytical columns with 10-32 PEEK end fittings and the Anion MicroMembrane Suppressor.

A. The Tefzel tubing provided by DIONEX has a 0.062" OD which is important so that the ferrule fits snugly on the tubing and thus makes a good seal. In order to easily slide the PEEK bolt and the ferrule onto the Tefzel tubing, a short section at one end of the tubing should be stretched slightly to reduce the its diameter. Cut the tubing in the middle of the stretched section.

B. Slide the end of the tubing with the reduced diameter through the hex-end of the PEEK bolt and then through the ferrule. The ferrule is symmetrical and can be placed onto the tubing through either end.

C. Pull the tubing through the 10-32 PEEK bolt and the ferrule until the ferrule fits snugly on a section of the unstretched 0.062" OD tubing.

D. Cut the tubing off approximately 1/16" past the end of the ferrule. Make sure that the end is cut off carefully at a right angle to the length of the tubing and that there are no nicks or burrs on the end of the tubing. Being careful while making liquid line fittings will reduce dead volumes and band broadening. This is important for maximum system performance.

E. Place the tubing with the ferrule and the 10-32 PEEK bolt into the 10-32 port and push the tubing through the bolt until it fits snugly against the bottom of the port. Making sure that there is no gap between the end of the tubing and the bottom of the port will ensure low dead volumes and minimal band broadening. This is important for maximum system performance.

F. Tighten the 10-32 PEEK bolt by hand until it is fingertight. As the bolt is tightened further, the compression of the ferrule will also compress the Tefzel tubing. Overtightening can reduce the ID of the tubing until flow through the tubing is completely restricted. Overtightening the bolt can also damage the mating surfaces of the 10-32 port. Fingertight torquing of the liquid line bolts provides leak free operation up to approximately 3000 psi.

G. For the best performance, end fittings should be mated to ports. Once a ferrule has been compressed inside a particular 10-32 port, it can be removed and reinstalled in the same port without sacrificing performance. If a liquid line needs to be routed to a new port, a new ferrule should be mounted on the end of the line and compressed in the new port to form a mated ferrule/port pair. This ensures the best sealing properties.

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APPENDIX A - MODIFICATION OF THE EDM-I

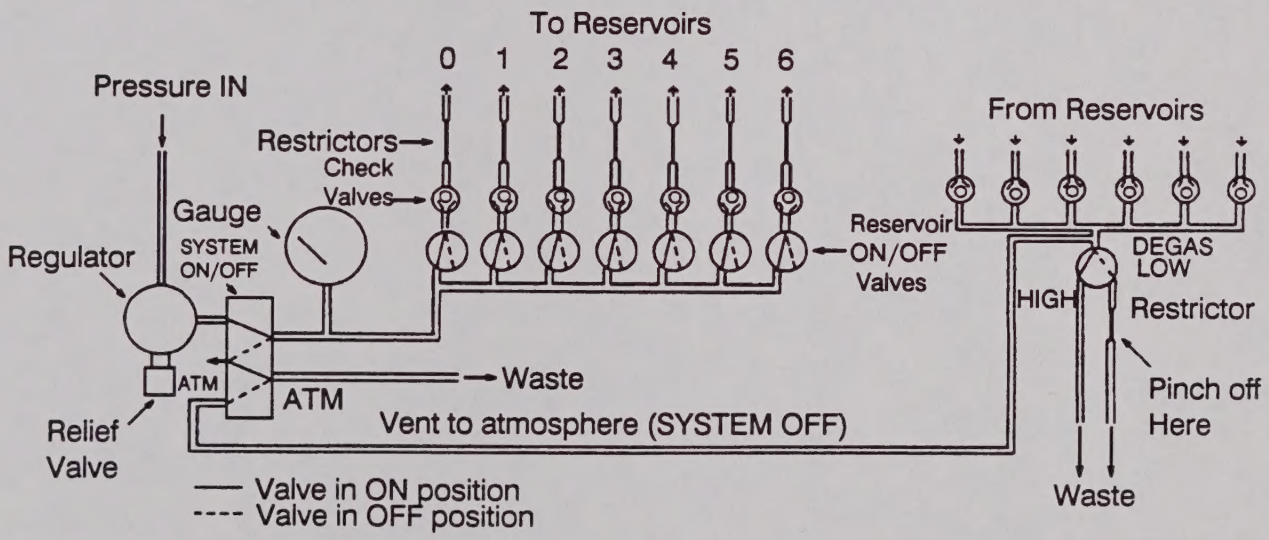
The EDM-I may be modified to eliminate continuous sparging when the HIGH/LOW toggle is in the LOW position. The reservoir can be purged with helium while the eluant is being prepared and the prepared eluant blanketed with helium.

A. Remove the EDM-I cover.

B. Locate the tubing connected to the low pressure side of the Degas Rate valve (Figure A-1). Fold it in half and push a coupler over the fold to cut off the air flow.

C. Install quick connectors (P/N 039167) in the vent lines returning from each reservoir to the EDM-I.

D. Remove the slip-on filter from the eluant outlet and the sparge line of each eluant reservoir. This prevents carbon dioxide absorption on the filter when refilling the reservoirs.



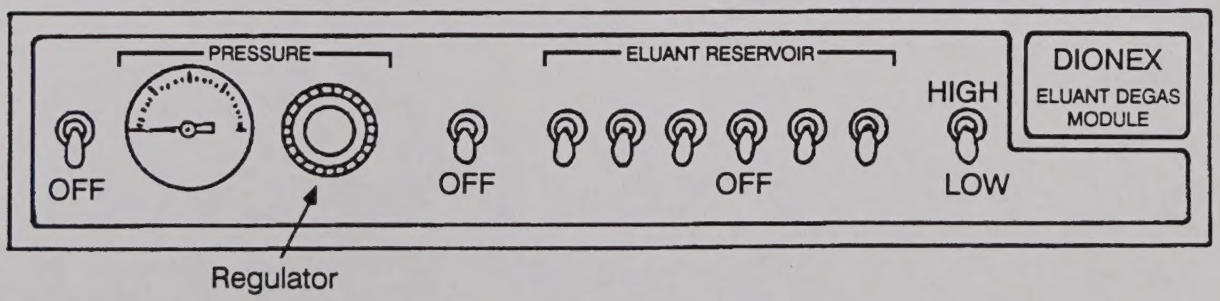
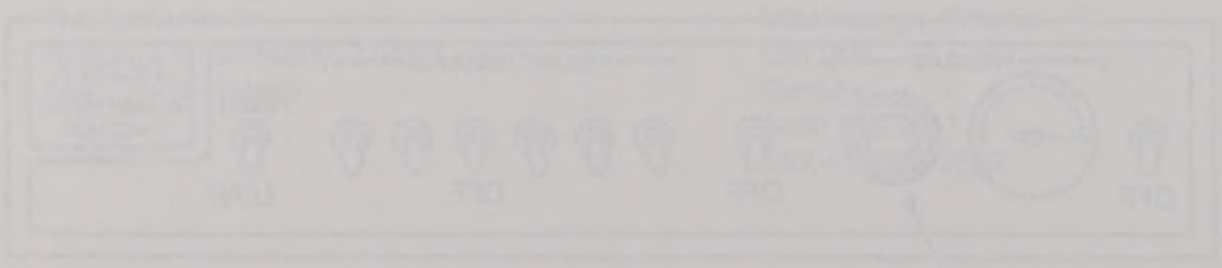


Figure A-1.



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